

# Optimization of Liquid Soap Preparation Formula with the Addition of Black Rice (Oryza Sativa L. Indica) Extract as Anti-Radical Free based Virgin Coconut Oil (VCO)

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**Abstract** - One way to prevent free radicals from entering the body is to bathe with soap. However, the commercial soap has negative impact to environment, because of chemical contains that disturb ecosystem. Black rice extract can be an antioxidant in soap because it contains anthocyanin pigments and has high bioactive compounds. Synthesis liquid soap that environment friendly was done by the saponification process between hydrolyzed VCO and black rice using bases KOH and NH4OH. This study aimed to analyze the effect of base concentrations, reaction time, and temperature treatments to quality of the liquid soap. The method uses Response Surface Methodology and the result tested by physicochemical test. The ratio of bases KOH:NH4OH (30:70, 50:50, 70:30)%, stirring time (65, 85, 105) minutes, and operating temperature (60, 75, 90) °C. Based on the results obtained, the optimum level of FFA was obtained with the minimized variable conditions, namely using a concentration of 16,4% KOH, and an operating temperature of 49,7 °C, a stirring time of 51.4 minutes, composite desirability of 0,89589, and FFA (%) Fit of 1,48958.

Keywords: liquid soap; virgin coconut oil; black rice; free fatty acids; saponification

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## **INTRODUCTION**

An imbalance in the body's number of antioxidants and free radicals can cause oxidative stress and trigger various degenerative diseases (WHO, 2016). One way to prevent free radicals from entering the body is to bathe with soap. Soap helps clean the body from germs and diseases caused by fungi and bacteria. The public prefers liquid soap to solid soap because it has economic value and is more effective (Sari et al., 2021). According to data from (Badan Pusat Statistik, 2019), the need for liquid soap in Indonesia has increased every year, namely in 2017, as much as 31,513 tons/year, in 2018, as many as 33,217 tons/year, and in 2019 as much as 34,071 tons/year.

Virgin Coconut Oil (VCO) is a raw material because it has the highest lauric acid content and helps

moisturize and smooth the skin (Afrozi et al., 2021). Hydrolysis is the reaction of the decomposition of oil or triglycerides with water as a reagent to obtain free fatty acids; the by-product is glycerol. The difference between VCO triglyceride fatty acids and VCO hydrolyzed fatty acids is that the hydrolyzed fatty acids in a free state are not bound to glycerol, which is called free fatty acids (Trivana & Karouw, 2017). The benefit of VCO hydrolysis is producing fatty acids and glycerol for oleochemicals such as soaps, cosmetics, and medicines (Setyopratomo, 2012).

Potassium hydroxide (KOH) is widely used for liquid soap because it is more soluble in water, while Ammonium hydroxide (NH<sub>4</sub>OH) is still rarely used, so research is needed to examine this. Many soaps on the market are soaps that use synthetic additives so that they can interfere with skin health. Meanwhile, the potential of organic black rice as a free radical scavenger containing anthocyanin pigments and a source of bioactive compounds has the potential as a source of antioxidants (Widyawati et al., 2014). According to (Ichikawa et al., 2001), the content of black rice, such as minerals, protein, vitamins, and fiber, is higher than white rice.

## METHODOLOGY

The primary raw materials used are hydrolyzed VCO, KOH, and NH<sub>4</sub>OH. The supporting ingredients are aquadest, glycerin, propylene glycol, essential oil, Coco-DEA, Phenolphthalein indicator, ethanol 96%, black rice extract, HCL, and vaseline.

The tools used are rotary vacuum evaporator, three-neck pumpkin, clamps and stands, thermometer, magnetic stirrer, return cooler, hose, electric stove, pot, burette, digital balance, watch glass, beaker glass, drop pipette, pH meter, Erlenmeyer, measuring cup, separator funnel, pycnometer, Ostwald viscometer, glass funnel, stirring rod, aluminum foil, stopwatch, and duster.

Table 1. Response Surface Methodology (RSM) Method							
Variable	Star Low	Low Point	Center Point	High Point	Star High		
Base ratio (%)	16,4:83,6	30:70	50:50	70:30	83,6:16,4		
Mixing time (minutes)	51,4	65	85	105	118,6		
Temperature °C	49,8	60	75	90	100,2		

## RESULTS AND DISCUSSION Results of Soap Physicochemical Analysis Table 2. Liquid Bath Soap Physicochemical Test Results

				*	pł	ł			Free	Free
Run	KOH	NH <sub>4</sub> OH	Time	Temperature			Density	Viscosity	Alkali	Fatty
Kull	(%)	(%)	(Minutes)	(°C)	Before	After	(gr/ml)	(cP)	(%)	Acid
									(70)	(%)
1	30	70	65	60	12	8	0,990	62,573	0	1,763
2	30	70	65	90	12	8	0,997	65,078	0	2,542
3	30	70	105	60	14	8	1,037	71,924	0	1,886
4	30	70	105	90	14	8	1,042	76,003	0	2,665
5	70	30	65	60	14	9	1,097	91,832	0	2,214
6	70	30	65	90	14	9	1,100	94,360	0	2,583
7	70	30	105	60	14	9	1,098	93,094	0	1,886
8	70	30	105	90	14	9	1,105	97,645	0	2,583
9	16,4	83,6	85	75	14	8	0,985	58,196	0	1,353
10	83,6	16,4	85	75	14	9	1,125	99,954	0	2,132
11	50	50	51,4	75	14	9	1,044	78,861	0	1,681
12	50	50	118,6	75	11	9	1,069	82,899	0	1,763
13	50	50	85	49,7	14	9	1,050	83,598	0	2,214
14	50	50	85	100,3	11	9	1,070	88,522	0	2,501
15	50	50	85	75	14	9	1,053	84.002	0	1 0 1 5
(C)	30	30	83	13	14	9	1,055	84,903	0	1,845
16	50	50	05	75	14	0	1.052	84.002	0	1 0 1 5
(C)	50	50	85	75	14	9	1,053	84,903	0	1,845

Determining the pH value aims to indicate the acidity and alkalinity of the resulting liquid soap. From the 16 runs that have been carried out, initially, the pH of the liquid soap ranged from 11-14, but after lowering the pH to 8-9. First, lower the pH of the soap, which can be done by adding acidic compounds, one of which is citric acid. Citric acid is an organic acid formed naturally in fruits, classified as a weak acid, and has a pH of 3-6 (Surianti et al., 2015). The pH value of soap is determined by the alkaline ingredients that make up the soap, namely KOH, which is a strong base, and NH4OH, which is a weak base. The factors that make the pH more

excellent are the amount of base added, the length of stirring time, and the temperature in making liquid soap formulas (Nurul et al., 2019). The acid mantle is the top layer and protective surface of the skin made of oil and sweat to keep the skin moist. The skin's pH must be maintained for it to work correctly and look healthy. If the pH of the soap is too alkaline, it causes dry and sensitive skin, while if it is too acidic, it causes the skin to become inflamed.

The density of liquid soap ranges from 0,990 - 1,125 grams/ml. Based on (SNI 3532-2016), the allowed density of liquid soap is 1.01 - 1.1 gram/ml. From the results of the study, it can be seen that the

soap variables that according to the soap quality requirements are variables 3, 4, 5, 6, 7, 8, 11, 12, 13, 14, 15, and 16. Adding KOH and NH4OH bases, time variations, and operating temperature can affect the density of soap, and the ratio is directly proportional. If the mass of KOH is greater than VCO, the density will be greater, and vice versa (Agusta et al., 2016). Because the density of KOH is 2.04 grams/ml, NH4OH is 0.88 grams/ml, while VCO is 0.92 grams/ml (Khairunisa, 2016).

Viscosity testing is carried out to determine the size of the viscosity of a substance in a fluid using a viscometer (Yulianti et al., 2015). The viscosity in the liquid soap experiment ranged from 58,196 cP to 99.954 cP. Based on SNI 3532-2016, the allowable viscosity of liquid soap is 400 - 4000 cP. If seen from the table above, no sample of liquid soap meets SNI. As the concentration of KOH increases, the length of time, and the high operating temperature, the product's viscosity will also increase (Agusta et al., 2016). Several things affect the viscosity of a product, namely the viscosity of the solvent, the contribution of the dissolved material, and the integration of the two (Wiyono et al., 2020). If the composition of the soapmaking ingredients (hydrolyzed VCO, KOH, and NH<sub>4</sub>OH) is more balanced, the saponification process runs ideally, so the resulting soap product is thicker (Nurul et al., 2019). An increase in the water ratio causes a decrease in viscosity.

Free alkali is not bound as a compound during soap making due to the addition of excess alkali during the saponification process (Gusviputri et al., 2017). The result of the free alkali test for liquid soap is 0% because there is no free alkali in liquid soap after adding other supporting materials and citric acid as a pH lowering agent. Therefore, free alkaline content is more dominant in soap paste than in liquid soap. Based on (SNI 3532-2016), the permissible value of alkali-free liquid soap is not more than 0.14% for soap based on KOH. All soap variables are in accordance with SNI 3532-2016 because the results are below 0.14%. Indicator Phenolphthalein (PP) is commonly used as an indicator for acid-base titrations. Colorless in acidic solutions and pink/pink in alkaline solutions. Adding this PP indicator is to prove that the sample is alkaline or acidic. KOH is used to measure some fatty acids that are free from oil. This analysis uses the acidimetric method because this method is a quantitative determination of the levels of compounds that are alkaline using acid standards (Rizky, 2013). The higher the concentration of KOH, the more alkaline the soap will be, so the quality is not good and causes itching. Soap, which usually contains high-free alkali, is used as laundry soap (Adelila Sari et al., 2019). The first factor for decreasing free alkali is the addition of given water because water can reduce the concentration of free alkali in soap (Wiyono et al., 2020).

Free fatty acids are acids that are not bound as compounds with sodium or triglycerides (neutral fats). A good soap is a soap produced from a perfect saponification reaction, hoping there will be no residue after the reaction (Fanani et al., 2020). The results of the free fatty acid test of liquid soap ranged from 1.353 to 2.583%. According to SNI 3532-2016, soap's free fatty acid content should not exceed 2.5%. Variables 1, 3, 5, 7, 9, 10, 11, 12, 15, 16 met the SNI standard. According to (Paramita et al., 2014), the greater the amount of oil compared to KOH in soap, the more free fatty acids increase. The presence of free fatty acids in soap can reduce soap's binding power to soap's binding capacity to dirt, oil, fat, or sweat (Agustini & Winarni, 2017). Fatty acids that are too high will affect the process of emulsifying soap with dirt and reduce the binding capacity of soap to dirt, oil, fat, and sweat (Ayu et al., 2010). These free fatty acids cannot bind dirt because they are polar, while dirt, fat, and sweat are non-polar.

#### Analysis of Response Surface Design of Free Fatty Acid in Liquid Soap

Tabel 3. Model Summary from Response Surface Regression for Free Fatty Acid

S	R-sq	R-sq(adj)	R-sq(pred)
0,344924	39,25%	24,06%	0,00%

Table 3 shows the model summary of the response surface regression. The data interpretation should be made by referring to the value of R-square adjust (R-aq(adj)) because the values tend to be constant and sensitive to changes in the independent variables. R square has a value between 0 - 1, with the provision that the closer to the number one, the better. But the R-square will change if you add another variable. The more independent variables used, the more bias or "noise" in the model, and this cannot be explained by R-square and is a weakness of R-square itself. An adjusted R square value is more suitable for

determining how the independent variable explains the dependent variable if the researcher uses more than two independent variables. This R squared adjusted into account the sample data and the number of variables used so that the R squared adjusted value does not always increase when additional variables are added. These results obtained an R square adjust of 24.06%, which means a 24.06% value of the dependent variable (%FFA of liquid soap), which is influenced by independent variables in the study (KOH base concentration, length of time, and operating temperature). From the analysis of the variance table, it can be seen in the linear part that the P-value of the KOH concentration is 0,202, where this value is more significant than ( $\alpha = 0.05$ ), which means the KOH concentration variable does not have a significant effect on the amount of %FFA Then the time variable shows a P-value of 0.966 where this value is more significant than ( $\alpha = 0.05$ ) which means that the time variable does not have a significant effect on the amount of FFA levels produced. Then the last is the temperature variable, which has a P-value of 0.031, which is smaller than ( $\alpha = 0.05$ ), which means that the temperature variable significantly affects the amount of %FFA produced.

Source	DF	Adj SS	Adj MS	<b>F-Value</b>	<b>P-Value</b>
Regression	3	0,92237	0,307457	2,58	0,102
X1 KOH (%)	1	0,21649	0,216493	1,82	0,202
X <sub>2</sub> Time (Minutes)	1	0,00023	0,000228	0,00	0,966
X <sub>3</sub> Temperature (°C)	1	0,70565	0,705649	5,93	0,031
Error	12	1,42767	0,118972		
Lack-of-Fit	11	1,42767	0,129788	*	*
Pure Error	1	0,00000	0,000000		
Total	15	2,3004			

Tabel 4. Analysis of Variance from Response Surface Regression of Free Fatty Acids (%)

In table 4, the analysis of variance obtained the P-value for the overall regression model of 0,102, where the value of this P-value is <0.05. This means that the response surface regression test is considered to have represented all of the existing data. From the analysis of the variance table, it can be seen in the linear part that the P-value for the KOH concentration is 0,202, where this value is more significant than ( $\alpha = 0.05$ ), which means that the KOH concentration variable does not have a significant effect on the amount of FFA levels obtained. First, generated. Then the time variable shows a P-value of 0,966, which is greater than ( $\alpha = 0.05$ ), which means that the time variable does not significantly affect the amount of

FFA levels produced. Then the last is the temperature variable, which has a P-value of 0,031 where this value is smaller than ( $\alpha = 0.05$ ), which means that the temperature variable significantly affects the amount of FFA levels produced.

The regression equation can be written as:

FFA (%) = 0.624 + 0.00630 KOH concentration (%) + 0.00020 Time (Minutes) + 0.01514 Temperature (°C)

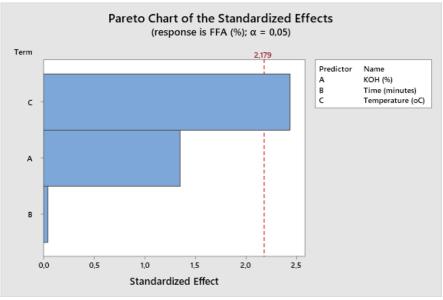


Figure 1. Pareto Diagram of the Standard Effect

The Pareto chart was created to help identify the significant factors that affect the free fatty acid levels resulting from the experiment. From Figure 2, it can be seen that the temperature variable has the most significant effect on the amount of FFA produced. Then, there is a KOH concentration variable that affects the amount of %FFA and a time variable with the slightest effect on the amount of %FFA levels.

After performing a regression analysis on the response surface design, it is also necessary to look at the residual plot of the resulting FFA levels. Residual is the difference between the dependent variable or Y with Y prediction. Y prediction is the value of Y based on the regression equation results. The residual plot can be observed in Figure 2.

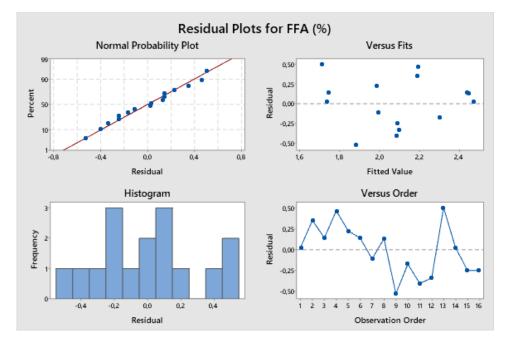


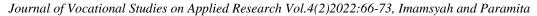
Figure 2. Graph of Residual Plots from Analysis of Independent Variables on Free Fatty Acid

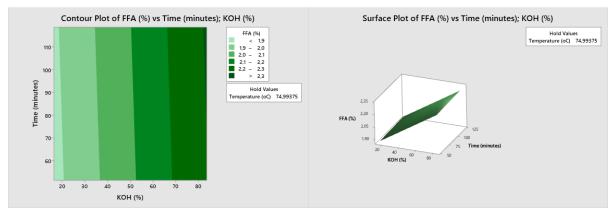
The histogram graph in Figure 2 shows that the histogram resembles a bell facing upwards, meaning that the residuals are said to contribute usually. A bell-shaped curve, also called a standard, or normal curve represents the normal distribution of test numbers or values. The normal distribution has a normal/directed pattern distribution, a mean of zero, and a standard deviation of one. This distribution is nicknamed the bell curve because the probability density function's graph is similar to a bell's shape. Therefore, data that is usually distributed will minimize the occurrence of bias.

In the normal probability plot graph, the plot results are not randomly distributed but follow a straight line, or the dots follow and approach the diagonal line so that it can be concluded that the regression model meets the assumption of normality and the residuals are normally distributed. The normality assumption test is a test carried out to assess the distribution of data in a group of data or variables and whether the data distribution is usually distributed. A good regression model has a normal or close to normal data distribution.

In the verses fit and versus order graphs, it can be concluded that there are no symptoms of heteroscedasticity because the plots spread evenly above and below the 0 axes without forming a particular pattern.

Furthermore, to analyze the relationship between variables, contour plots and surface plots can be used to analyze the effect of time and concentration variables on %FFA with temperature and concentration variables on %FFA. Analyze the effect of time and concentration variables on %FFA can be seen in Figure 3.





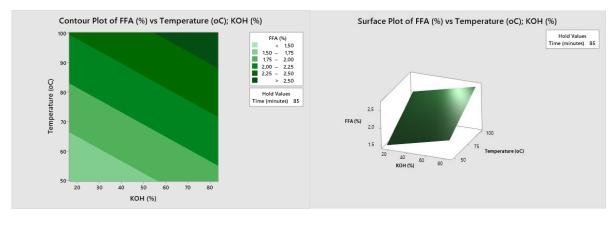
(a) Contour Plot



Figure 3. Analysis of the Effect of FFA (%) vs. Time (Minutes); KOH Concentration (%)

In Figure 4 in the contour plot section, it can be seen that the most optimum part (producing trim levels of FFA) is marked with a light green color and is present at 50-60 minutes, and the concentration of KOH is 15-20%. Then on the surface plot, the most optimum level of FFA is shown at the top end of the curve, which is also at 50 minutes and 20% KOH concentration. The greater the time, the greater the FFA levels produced, while the smaller the time, the

smaller the FFA levels. Then for the concentration of KOH, the greater the concentration of KOH added, the higher the levels of FFA formed, and vice versa, the smaller the concentration of KOH, the lower the levels of FFA. Then, the effect of time and concentration variables on FFA levels with temperature and concentration variables on FFA levels can be seen in Figure 4.



(a) Contour Plot

(b) Surface Plot

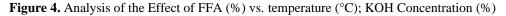


Figure 4 in the contour plot shows that the most optimum part (producing trim levels of FFA) is marked with a light green color and is present at a temperature of 50 °C and a 20% KOH concentration. Then on the surface plot, the most optimum level of FFA is shown at the top end of the curve, which is also at 50 °C and 20% KOH concentration. The greater the

temperature, the higher the FFA content produced, while the lower the temperature, the smaller the FFA content. Then for the concentration of KOH, the greater the concentration of KOH added, the higher the levels of FFA formed, and vice versa, the smaller the concentration of KOH, the lower the levels of FFA.

Table 5. Solution of Independent Variable Free Fatty Acid Content in Soap Making

Solution	KOH Concentration(%)	Time (Minutes)	Temperature (°C)	FFA (%) Fit	Composite Desirability
1	16,4	51,4	49,7	1,48958	0,895897

Based on the results obtained, the optimum level of FFA (resulting in a small %FFA) was obtained with the minimum variable conditions is variable 9, which are listed in Table 4.9, using a KOH concentration of 16.4%, an operating temperature of 49.7 °C, and a stirring time of 51. 4 minutes. As a result, the program found the optimum value with

## CONCLUSIONS

Determining the best solution for the soap variable with the lowest FFA content using Minitab 19 software and falling on the 9th variable, namely the minimize variable, where the KOH concentration is 16.4%, the stirring time is 51.4 minutes, and the operating temperature is 49.7 °C. Variable soap 9 has

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Badan Pusat Statistik. (2019). Industri Sabun dan Bahan Pembersih Keperluan Rumah Tangga. composite desirability of 0.895897 and FFA (%) Fit of 1,48958. The desirability value is the value of the optimization objective function, which indicates the program's ability to fulfill the wishes based on the specified criteria. The range of desirability values is 0-1.0. This shows that the closer the value to 1.0, the more perfect (Nurul et al., 2019).

a pH of 8, a free alkali content of 0.112%, and a free fatty acid content of 1.353%. The independent variable analysis found that the lower the KOH concentration, the longer the stirring time and the operating temperature, and the lower the free fatty acid content.

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